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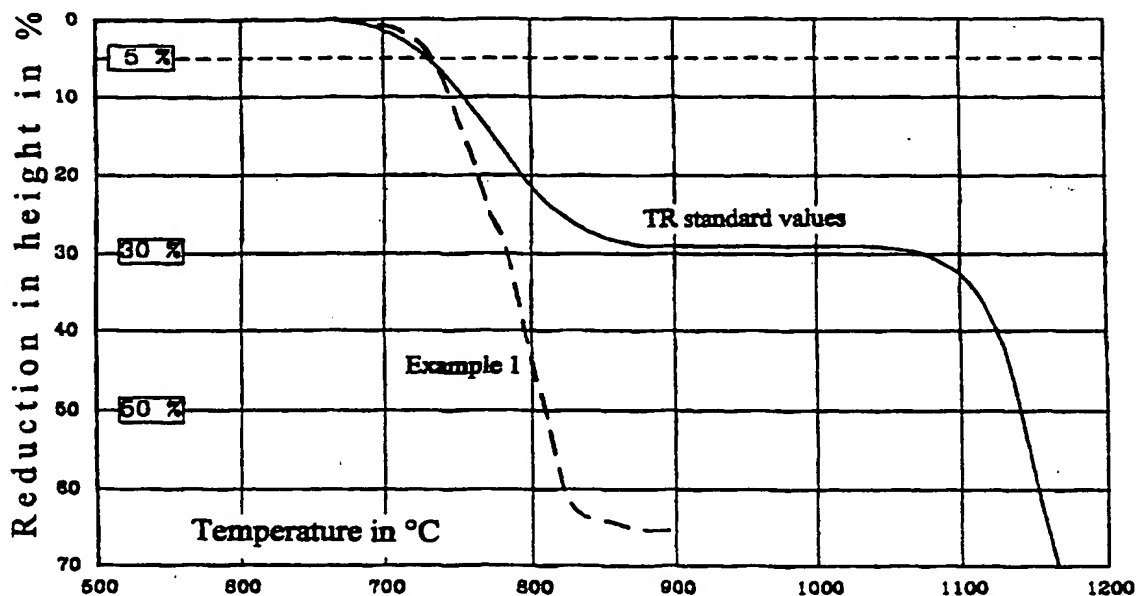
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(54) Title: A MINERAL-FIBER COMPOSITION

Temperature behaviour (Swedish method)



(57) Abstract

A biologically degradable mineral-fiber composition characterized by the following constituents in percent by weight: SiO₂: 40 to less than 52; Al₂O₃: less than 4; CaO: more than 25 and up to 45; MgO: 5 to 15; BaO: 0 to 7; Na₂O: 2 to 12; K₂O: 0 to 10; Na₂O + K₂O: 2 to 15; TiO₂, Fe₂O₃, MnO: 0 to 5.

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A mineral-fiber composition

The present invention relates to a mineral-fiber composition that is biologically degradable.

The prior art describes some mineral-fiber compositions which are said to be biologically degradable.

The biological degradability of mineral-fiber compositions is of great importance because various studies point out that mineral fibers with very small diameters in the range of less than 3 microns are suspected to be carcinogenic, while biologically degradable mineral fibers of such dimensions show no carcinogenicity.

However the mineral fiber compositions must also be easy to process by known methods for making mineral wool with a small diameter, in particular the jet process. This means in particular a sufficient processing range of for example 80°C and suitable viscosity of the glass melt.

Also, the mechanical and thermal properties of the mineral fibers, or the products produced therefrom, are of crucial importance. For example mineral fibers are used to a great extent for insulation purposes. Particularly for use in the industrial sector, sufficient temperature resistance of the mineral fibers is necessary.

The invention is based on the problem of providing a novel mineral-fiber composition that is characterized by biological degradability, has good temperature resistance and is easy to process.

The invention is based on the finding that this problem can be solved by a mineral-fiber composition that consists substantially of silicon dioxide and alkaline-

earth oxides, and also contains sodium oxide and/or potassium oxide as a melting accelerator and a sizable amount of aluminum oxide to increase the temperature resistance.

It has turned out that such mineral-fiber compositions fulfill the combination of the necessary properties, namely biological degradability, sufficient temperature resistance for insulated objects in industry, and good processibility for producing the mineral wool as such and the products. This means at the same time that the upper devitrification temperature of the melt is preferably under 1300°C. The mean fiber diameter is preferably 10 microns or less, and is in particular between 2.5 and 5 microns.

The object of the invention is a mineral-fiber composition that is biologically degradable, characterized by the following constituents in percent by weight:

SiO ₂	40 to less than 52
Al ₂ O ₃	less than 4
CaO	more than 25 and up to 45
MgO	5 to 15
BaO	0 to 7
Na ₂ O	2 to 12
K ₂ O	0 to 10
Na ₂ O + K ₂ O	2 to 15
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 5.

The inventive mineral-fiber compositions are in particular easy to draw by the jet process, i.e. one obtains a fine, low-slug mineral wool.

The mineral fibers reach a high temperature resistance of at least 730°C.

The mineral fibers show high biological degradability.

The addition of sodium oxide and/or potassium oxide lowers the melting point, thereby improving processability in the melting process. Also, if the mineral-wool composition contains sodium it is advantageous to use up to 35% broken waste glass.

The inventive mineral-fiber compositions can preferably be melted at melting temperatures of 1350 to 1450°C in melting chambers fired by fossil fuels, in particular natural gas. Such melting chambers can produce a homogeneous melt, which is the precondition for constant product quality. Homogeneity of the glass melt also facilitates reproducibility of the fiber-forming process and thus the thermal and mechanical product properties. Furthermore, the constant chemical composition of the thus produced mineral wool leads to controllable biological degradability.

In particular the addition of aluminum oxide increases the temperature resistance of the mineral wool.

The inventive mineral-fiber compositions preferably have the following constituents in percent by weight:

SiO ₂	40	to 51.5
Al ₂ O ₃	2 to less than 4	
CaO	25.5	to 40
MgO	8	to 15
BaO	0	to 5
Na ₂ O	3	to 8
K ₂ O	2	to 10
Na ₂ O + K ₂ O	5	to 10
TiO ₂ , Fe ₂ O ₃ , MnO	0	to 3.

A content of silicon oxide in the range of 40 to 55 percent by weight is particularly preferred.

With respect to the alkali oxides a range of 5 to 8 percent by weight is particularly preferred. Aluminum oxide is preferably present in a range between 3 and 4 percent by weight.

Barium oxide, which can be used instead of calcium or magnesium oxide, is present in an amount up to 7 percent by weight, preferably up to 5 percent by weight, in particular 0.5 to 3 percent by weight.

Sodium oxide is preferably present in an amount of more than 2 percent by weight.

An aluminum oxide content between 1 and 2 percent by weight, in particular of 1.5 percent by weight, is also particularly preferred.

The content of iron is preferably 0.5 to 2.5 percent by weight.

To assess biological degradability the standard powder test of the German Glass Society was used. This is an easily conducted method and gives a sufficient measure of biological degradability when used with a simulated physiological lung fluid at 37°C. The method is described in L. Springer, "Laboratoriumsbuch für die Glasindustrie", 3rd edition, 1950, Halle/S: W. Knapp Verlag.

The temperature behavior of the mineral fibers was determined by the "Swedish method". In this method a silit tube furnace is used with a horizontal working tube open on both sides having a length of 350 mm and an inside diameter of 27 mm. In the center of the furnace there is a small

ceramic supporting plate (30 X 20 X 3 mm) for holding the test sample. The test sample has dimensions of 12 X 12 X 12 mm or 12 mm \varnothing X 12 mm height. The bulk density is normally 100 kg/m³. The temperature increase is 5 K/min. The change in test sample height is determined continuously with a reading optic.

The invention shall be described in more detail in the following with reference to examples.

Example 1

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	51
Al ₂ O ₃	3
Fe ₂ O ₃	0.3
CaO	31
MgO	10
Na ₂ O	5
K ₂ O	0.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.0 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

An investigation according to the standard powder test of the German Glass Society yielded a value of 40 mg/kg and thus a value for high biological degradability.

Determination of temperature behavior by the Swedish method yielded a temperature resistance at 5% reduction in height of 735°C, which can be clearly seen in the

corresponding diagram shown by way of example in the single drawing.

Example 2

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	46.5
Al ₂ O ₃	3.5
CaO	35
MgO	10
Na ₂ O	5.

This composition could also be processed well to mineral fibers with a mean diameter of 2.0 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

An investigation according to the standard powder test of the German Glass Society yielded a value of 35 mg/kg and thus a value for high biological degradability.

Determination of temperature behavior by the Swedish method yielded a temperature resistance at 5% reduction in height of 800°C.

This example shows that glass with a high aluminum oxide content has excellent temperature resistance, which is in turn a criterion for the fire resistance of the products.

Example 3

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	50
Al ₂ O ₃	2.8
Fe ₂ O ₃	0.9
CaO	25.6
MgO	9.7
Na ₂ O	4.9
K ₂ O	1.0
BaO	4.9.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Example 4

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	51
Al ₂ O ₃	2.6
Fe ₂ O ₃	1.1
CaO	27.9
MgO	10.4
Na ₂ O	3.2
K ₂ O	0.6
BaO	3.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Example 5

A mineral wool was produced with the following composition in percent by weight:

SiO ₂	50.9
Al ₂ O ₃	2.6
Fe ₂ O ₃	1.1
CaO	30
MgO	10.4
Na ₂ O	2.2
K ₂ O	1.6
BaO	1.1.

This composition could be processed well to mineral fibers with a mean diameter of 2.5 to 10 microns by the jet process at a drawing temperature between 1300 and 1400°C.

Claims

1. A mineral-fiber composition that is biologically degradable, characterized by the following constituents in percent by weight:

SiO ₂	40 to less than 52
Al ₂ O ₃	less than 4
CaO	more than 25 and up to 45
MgO	5 to 15
BaO	0 to 7
Na ₂ O	2 to 12
K ₂ O	0 to 10
Na ₂ O + K ₂ O	2 to 15
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 5.

2. The mineral-fiber composition of claim 1, characterized by the following constituents in percent by weight:

SiO ₂	40 to 51.5
Al ₂ O ₃	2 to less than 4
CaO	25.5 to 40
MgO	8 to 15
BaO	0 to 5
Na ₂ O	3 to 8
K ₂ O	2 to 10
Na ₂ O + K ₂ O	5 to 10
TiO ₂ , Fe ₂ O ₃ , MnO	0 to 3.

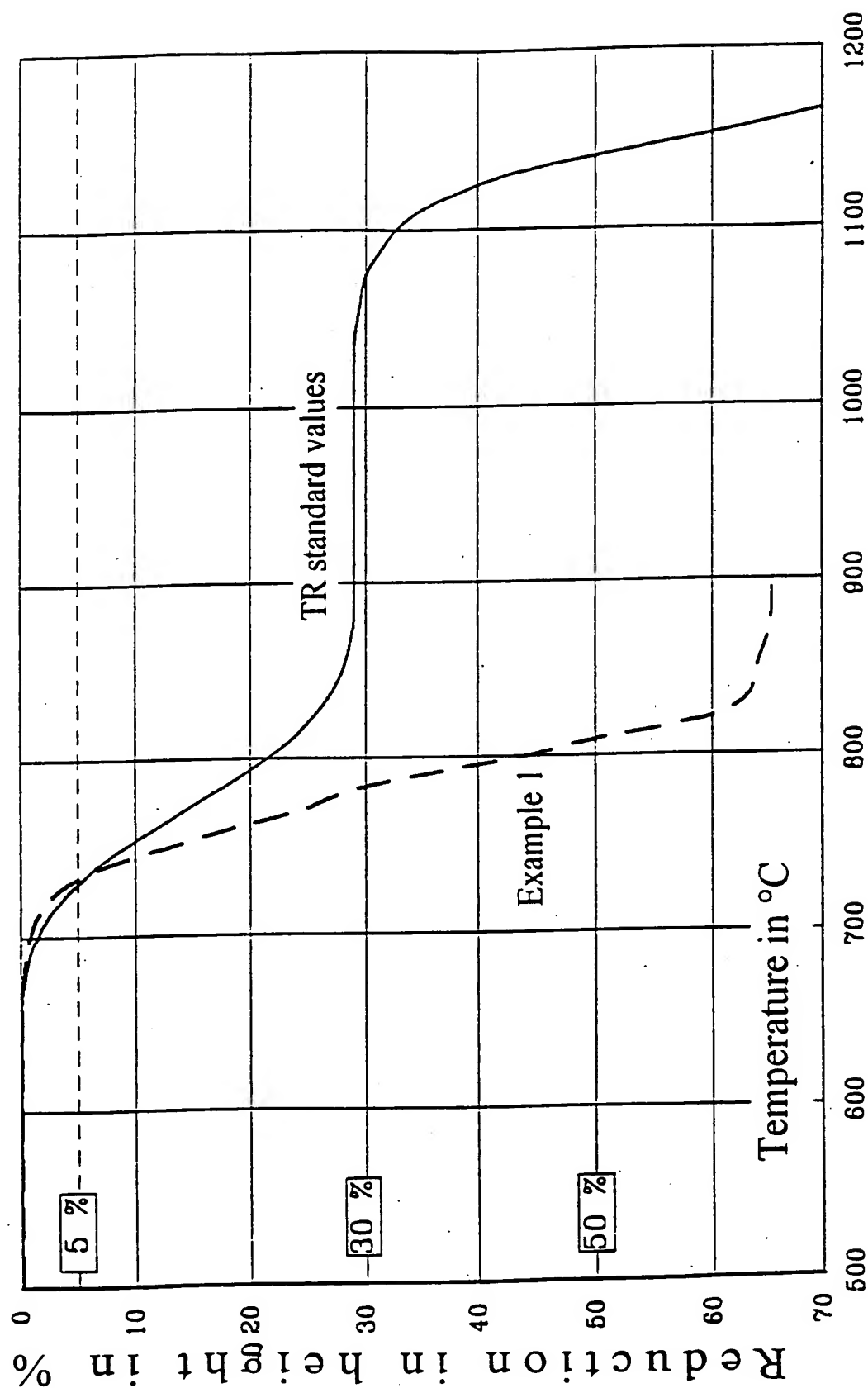
3. The mineral-fiber composition of claim 1 or 2, characterized in that the alkali oxides are present in an amount of 5 to 8 percent by weight.

4. The mineral-fiber composition of any of claims 1 to 3, characterized in that aluminum oxide is present in a content between 3 and 4 percent by weight.

5. The mineral-fiber composition of any of claims 1 to 4, characterized in that the content of iron is 0.5 to 2.5 percent by weight.

6. The mineral-fiber composition of any of claims 1 to 5, characterized in that the content of barium oxide is 0.5 to 4 percent by weight.

Temperature behaviour (Swedish method)



INTERNATIONAL SEARCH REPORT

Internal Application No
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IPC 6 C03C13/00 C03C13/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	FR,A,2 690 438 (ISOVER SAINT-GOBAIN) 29 October 1993 see page 1, line 37 - page 3, line 21 ---	1-6
X	WO,A,92 09536 (PAROC OY AB) 11 June 1992 see page 1, line 35 - page 4, line 3 ---	1-6
X	EP,A,0 459 897 (ISOVER SAINT-GOBAIN) 4 December 1991 see column 2, line 11 - column 3, line 15 -----	1-6

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Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FR-A-2690438	29-10-93	AU-B- 4263293	29-11-93
		BR-A- 9305492	11-10-94
		CA-A- 2110998	11-11-93
		CN-A- 1078708	24-11-93
		CZ-A- 9302865	19-10-94
		EP-A- 0596088	11-05-94
		WO-A- 9322251	11-11-93
		HU-A- 67212	28-03-95
		JP-T- 6508600	29-09-94
		NO-A- 934725	20-12-93
		SI-A- 9300218	31-12-93
WO-A-9209536	11-06-92	FI-B- 93346	15-12-94
		AT-T- 117662	15-02-95
		AU-A- 8908791	25-06-92
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EP-A-459897	04-12-91	FR-A- 2662688	06-12-91
		AT-T- 121378	15-05-95
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		CA-A- 2043699	02-12-91
		CN-A- 1059135	04-03-92
		DE-D- 69108981	24-05-95
		JP-A- 4228455	18-08-92
		US-A- 5250488	05-10-93